

**THERMAL CONTROL/OXIDATION RESISTANT COATINGS
FOR TITANIUM-BASED ALLOYS**

Ronald K. Clark and Terryl A. Wallace
NASA Langley Research Center
Hampton, VA

George R. Cunningham
Lockheed Missiles and Space Company
Palo Alto, CA

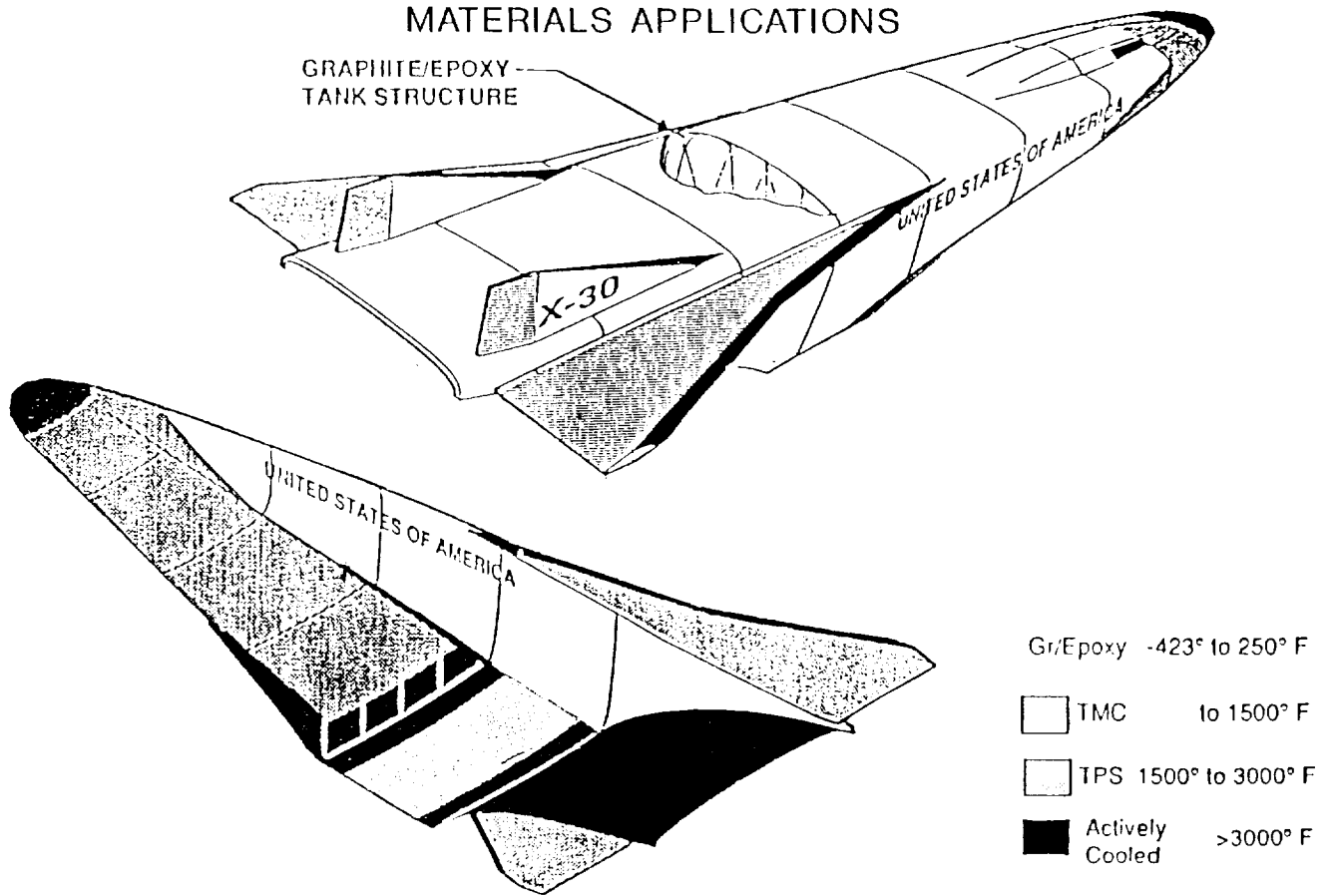
Karl E. Wiedemann
Analytical Services and Materials, Inc.
Hampton, VA

INTRODUCTION

Titanium-based alloys are candidate materials for hot structures and heat shields in hypersonic vehicles because of their low weight and high strength at elevated temperature. The figure shows a schematic diagram of the NASP vehicle configuration with regions for use of various materials systems mapped. Titanium-matrix composite (TMC) material, projected for use at temperatures to 1500°F, is shown over a significant portion of the vehicle. However, the range of conditions where they can be used is limited by their susceptibility to oxidation and loss of mechanical properties at high temperature. Protective coatings that shield these materials from oxidation will enable their use at higher temperatures. Further, coatings that provide a high emittance and low catalytic efficiency surface can significantly extend the applicability of these materials by reducing the net heating to the vehicle surfaces in hypersonic flight environments that contain dissociated species. The low catalytic efficiency reduces heat input from recombination of dissociated species in the environment and the high emittance results in greater heat flux rejection by radiation at the surface.

NASP VEHICLE CONFIGURATION

MATERIALS APPLICATIONS



THERMAL CONTROL/OXIDATION RESISTANT COATINGS FOR TITANIUM-BASED ALLOYS

Extensive research and development efforts have been expended toward development of thermal control and environmental protection coatings for NASP and generic hypersonic vehicle applications. The objective of the coatings development activities summarized here was to develop light-weight coatings for protecting advanced titanium alloys from oxidation in hypersonic vehicle applications. A number of new coating concepts have been evaluated. Coated samples were exposed to static oxidation tests at temperatures to 1000°C using a thermogravimetric apparatus. Samples were also exposed to simulated hypersonic flight conditions for up to 10 hr to determine their thermal and chemical stability and catalytic efficiency. The emittance of samples was determined before and after exposure to simulated hypersonic flight conditions.

Objective:

- Develop light-weight coatings for protecting advanced titanium alloys from oxidation in hypersonic applications
 - Thermal control thru high emittance and low catalysis
 - Oxidation protection thru oxygen barriers

Approach:

- Explore innovative new coating concepts/chemistries
- Conduct static/dynamic oxidation tests
- Determine heat transfer and oxidation characteristics

ENVIRONMENTAL RESISTANT COATINGS FOR TITANIUM-BASED ALLOYS

Environmental coatings provide protection of the substrate alloy by way of an oxygen barrier layer that retards oxidation and by the high emittance and low catalysis surface properties which lower the operating temperature for a given environment. In addition to these factors, successful coatings must be thermally and chemically stable and must be thin to minimize weight.

A variety of approaches to providing environmental coatings for hypersonic vehicle applications have been considered. Some approaches, such as pigmented paints, slurry coatings, and conversion coatings, have been abandoned because of thickness or weight considerations. Physical vapor deposition has limited potential for producing good coatings because of the difficulty in forming thin defect-free coatings. Two approaches that have good potential for forming micrometer-thick protective coatings are based on chemical vapor deposition (CVD) and sol-gel processes. Both of these methods are utilized for coatings development at NASA Langley Research Center (LaRC). CVD is being utilized in coatings development by Lockheed Missiles and Space Company under contract to NASA LaRC and sol-gel processes are being utilized for coatings research and development in the laboratory at LaRC.

Performance Requirements:

- Oxidation Protection
- Thermal/Chemical Stability
- Minimum Weight
- Low Catalytic Efficiency
- High Emittance

Approaches Considered:

- Pigmented Paints - Too Thick
- Slurry Coatings - Too Thick
- Reactive Coatings - Too Thick, Consume Substrate
- Physical Vapor Deposition - Difficult to Form Thin, Defect-Free Coating
- Chemical Vapor Deposition - Good Potential
- Sol-Gel - Good Potential

MATERIAL PROPERTIES THAT AFFECT STEADY STATE TEMPERATURE

The equilibrium temperature of a vehicle subjected to aerothermal heating is governed by the net rate of heat transfer to the vehicle, which is the difference between the heat input and the heat out. Heat input consists of two components: a convection heating term (q_{conv}) and a chemical recombination heating term ($q_{\text{catalytic}}$). The chemical recombination heating results when dissociated gas species (oxygen and nitrogen) present in the flow-field boundary layer recombine at the vehicle surface to form diatomic molecules. Heat is rejected (q_{out}) by the vehicle through radiative heat transfer. Thus the two material properties that have direct effect on the steady state temperature of a vehicle subject to aerothermal heating are catalytic efficiency of the surface and the hemispherical emittance of the surface: a low catalytic efficiency reduces the vehicle heating due to chemical recombination and a high emittance increases the heat rejection at the vehicle surface.

$$q_{\text{net}} = q_{\text{conv}} + q_{\text{catalytic}} - q_{\text{out}}$$

$$q_{\text{catalytic}} \propto \gamma \cdot h_R$$

$$q_{\text{out}} \propto \sigma \cdot \epsilon \cdot T^4$$

WHERE

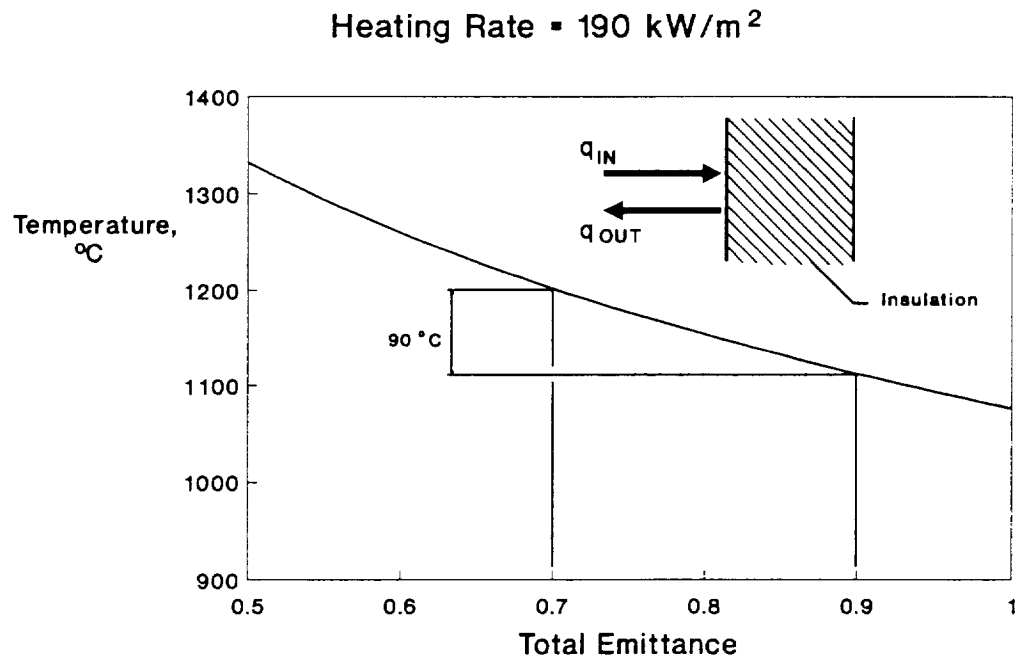
γ CATALYTIC EFFICIENCY

h_R HEAT OF RECOMBINATION

ϵ HEMISPHERICAL EMITTANCE

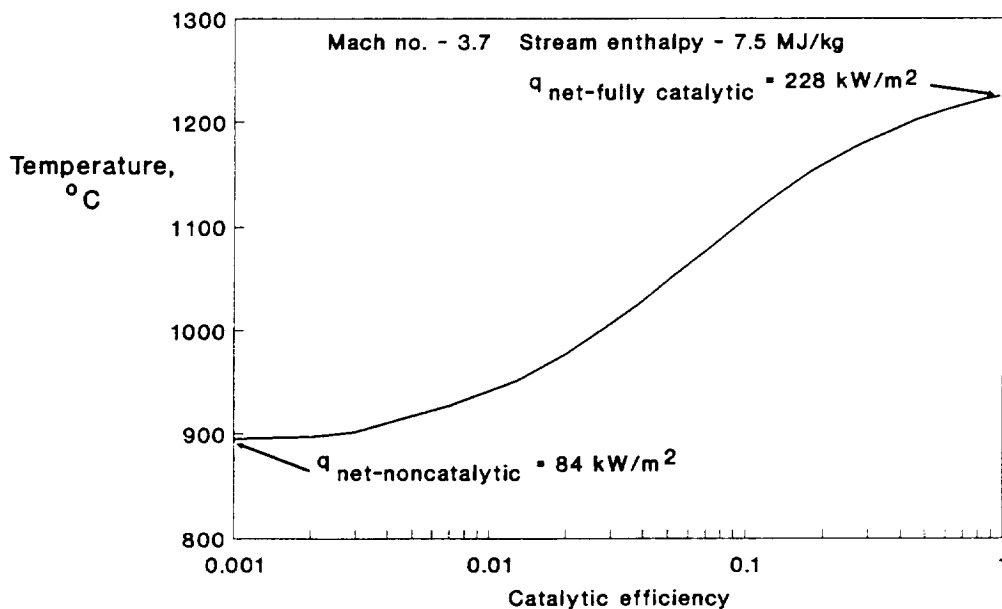
EFFECT OF EMITTANCE ON TEMPERATURE

The effect of emittance on surface temperature is demonstrated by the variation in equilibrium temperature with surface emittance for an insulated surface subjected to a constant heating rate. The figure shows a decrease of 90°C in temperature as the emittance increases from 0.7 to 0.9.



EFFECT OF CATALYTIC EFFICIENCY ON TEMPERATURE
TOTAL EMITTANCE - 0.8

The figure shows the variation in equilibrium temperature with catalytic efficiency for an insulated surface of 0.8 emittance exposed to a mild aerothermal environment (Mach no. 3.7 and 7.5 MJ/kg stream enthalpy). At very low catalytic efficiency levels the aerothermal heating is almost all due to convection heat transfer. At high catalytic efficiency levels, the total heating is more than double the convection heat transfer value. The figure shows that, for the very mild condition of this example, the impact of catalytic heating is to increase the equilibrium temperature by several hundred degrees.

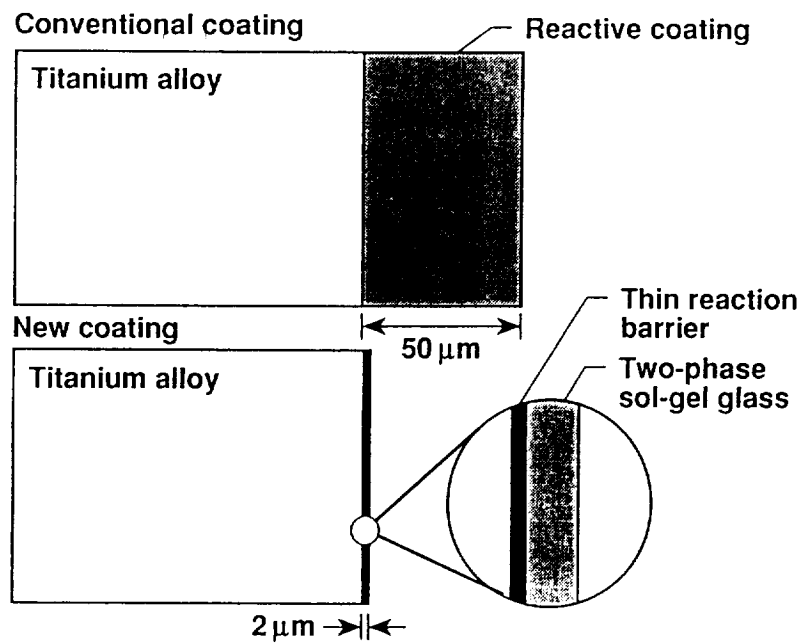


CONVENTIONAL REACTIVE COATING VERSUS THIN MULTI-LAYER GLASS COATING

Conventional coatings that provide good oxidation protection to metallic materials in high temperature oxidizing environments are commercially available. Conventional coatings on titanium-based alloys are typically formed by applying a slurry mixture to the surface and curing them in a furnace at high temperature. During the cure cycle the coating compound reacts with the alloy to form an intermetallic layer, which may be 25 to 50 μm in thickness. Concerns with use of this type coating include: reactive coatings consume substrate material as the coatings are formed, thick coatings represent a substantial weight penalty, and a very low ductility intermetallic layer at the surface may reduce the fracture toughness of the alloy.

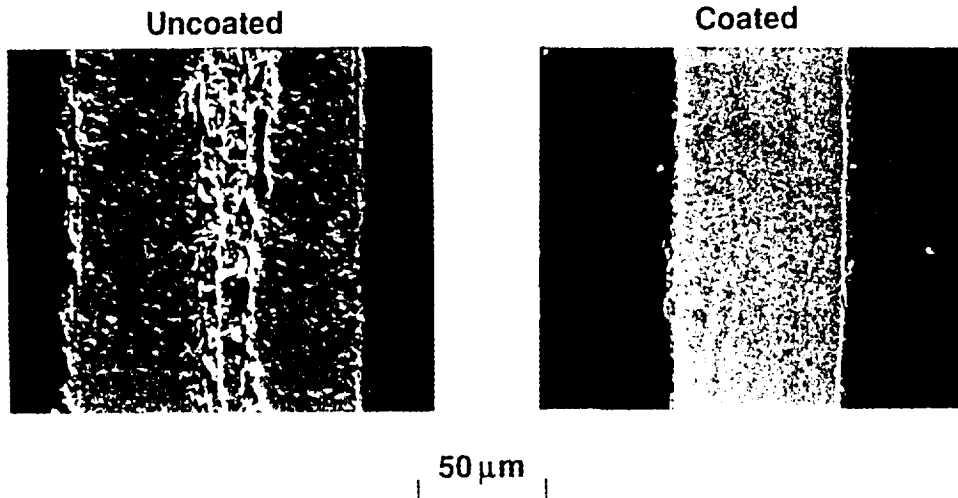
Development of new coatings was undertaken to overcome the limitations of conventional coatings. The focus of the coating research at LaRC was on achieving stable, thin, oxidation and/or thermal control coatings. Materials such as SiO_2 and Al_2O_3 , which have a low oxygen diffusivity, tend to react with titanium at high temperatures so the coating design includes multiple layers: a thin reaction barrier layer separating the alloy from a self-healing diffusion barrier layer. One form of diffusion barrier layer is shown in the figure: a two-phase glass wherein one phase becomes molten at high temperature and provides healing of any defects that form in the coating.

LaRC advances: Thin protective coatings from two-phase sol-gel glasses



CROSS-SECTIONAL MICROGRAPH OF CP TITANIUM
Static oxidation, 1 hr at 982°C

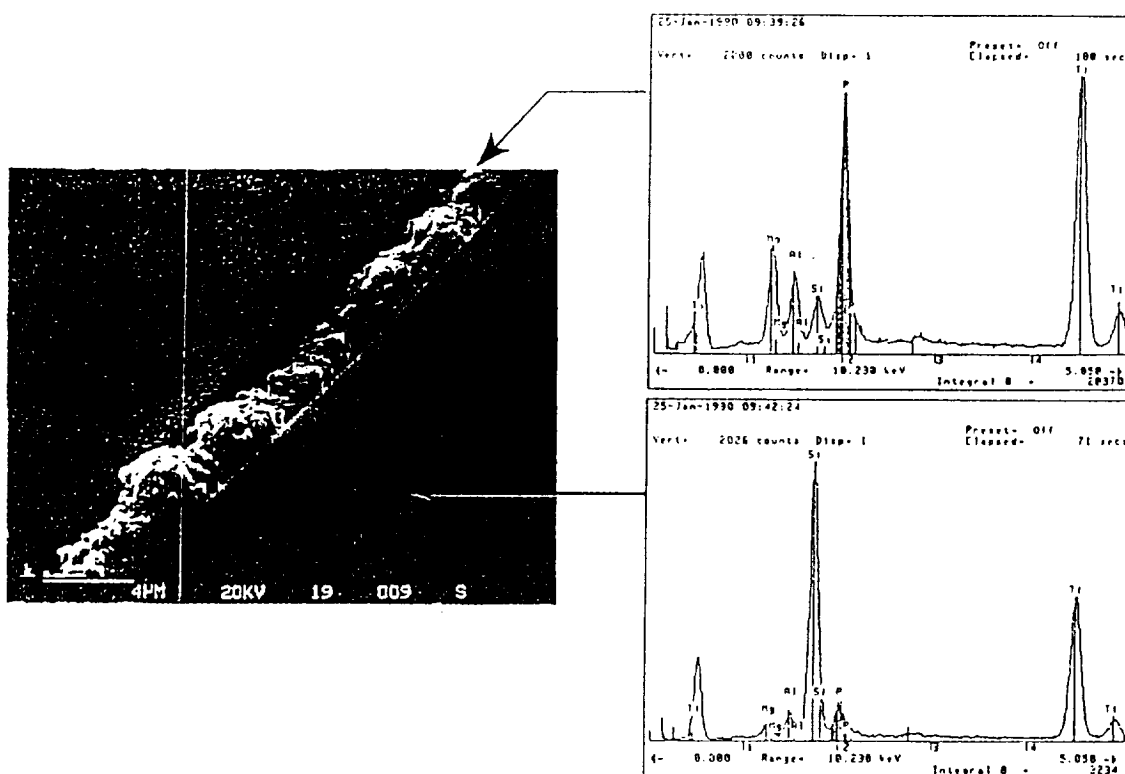
Many of the developmental activities related to sol-gel coatings technology were carried out using commercial purity titanium, which is highly reactive with oxygen at elevated temperature, so that coating imperfections and defects are easily observed. The figure shows cross-sectional micrographs of coated and uncoated titanium samples after static oxidation for 1 hr at 982°C. The uncoated sample was fully oxidized during that exposure, but the sample coated with a reaction barrier plus a two-phase glass shows no evidence of oxidation.



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**MICROGRAPH OF SURFACE OF COATED TITANIUM:
LOW MELT P-RICH PHASE FILLS CRACKS IN COATING
Oxidized 1 hr at 982°C**

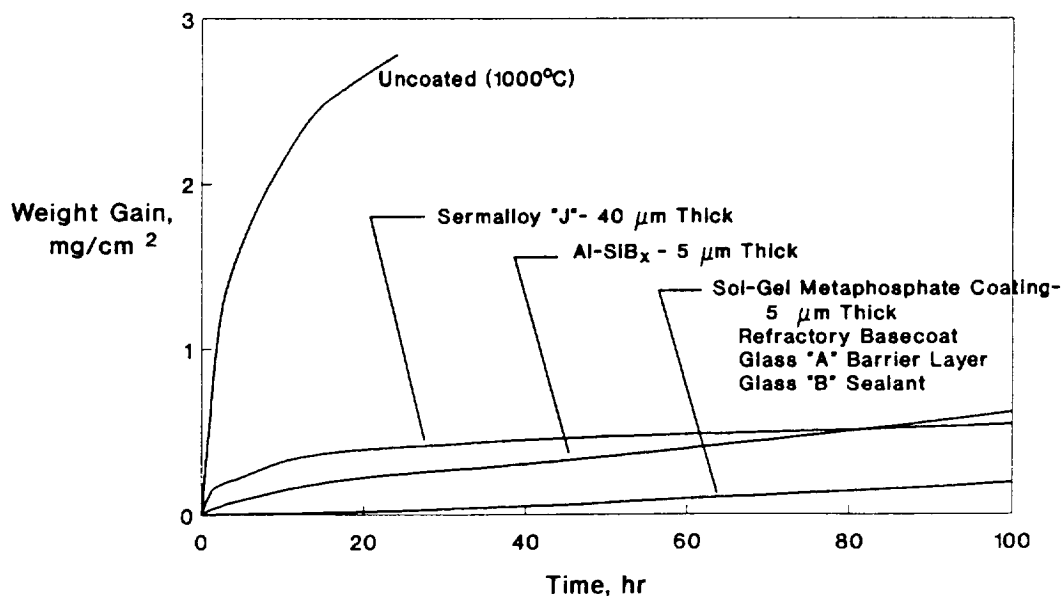
The figure contains a micrograph of the surface of a coated titanium sample that has been oxidized for 1 hr at 982°C. The surface is traversed by a 2- μm wide crack. Elemental analysis of the surface region of the crack showed the region to be phosphorus-rich, while the region distant from the crack is primarily silicon. These results provide graphic evidence of how the two-phase glass coating performs: microcracks that form in the glass coating are sealed by the lower melting phosphorus-rich glass phase which flows into the cracks to fill them. The two-phase glass coating is, therefore, self-healing.



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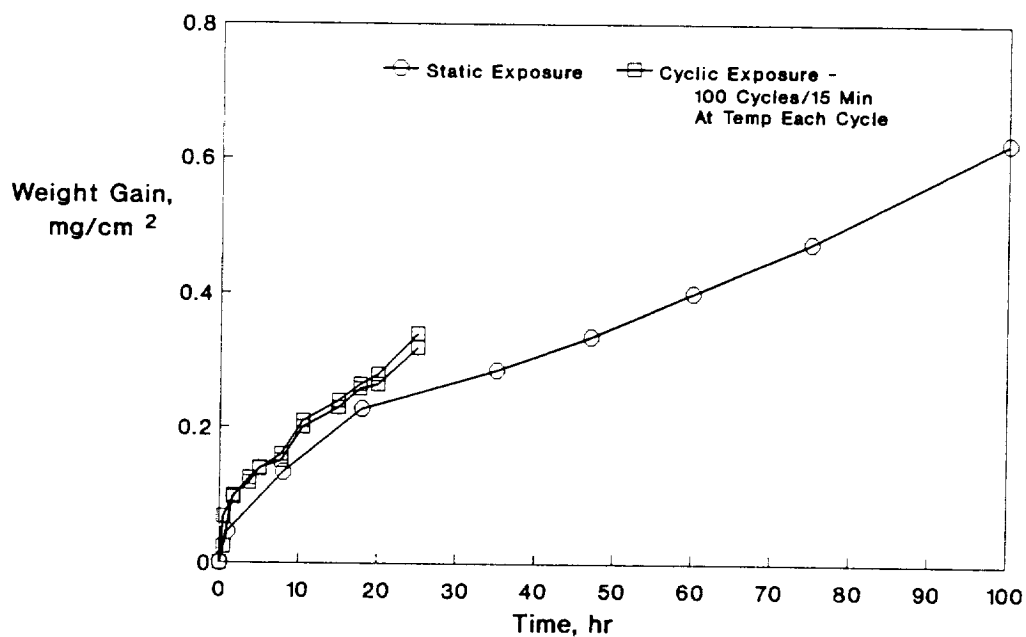
OXIDATION PROTECTIVE COATINGS STATIC OXIDATION OF Ti-14Al-21Nb AT 982°C

One assessment of the performance of oxidation protective coatings is obtained from static oxidation tests using a thermogravimetric apparatus that measures the weight change of a sample with time of exposure to oxidizing conditions. The figure shows weight gain for 982°C-100 hr static oxidation tests of Ti-14Al-21Nb samples with no coating and coated with Sermalloy "J", Al-SiB_x, and a multi-layer sol-gel coating. The Sermalloy "J" coating is a commercially available diffusion-type coating that measures about 40 μm in thickness. Because it performs very well as an oxidation protection coating it is included here as a reference for evaluating other coatings. The Al-SiB_x coating is a proprietary coating of Lockheed Missiles and Space Company. It is about 5 μm thick and is applied by physical vapor deposition (PVD) and chemical vapor deposition (CVD). Two 2 μm-thick layers of SiB_x (applied by CVD) are separated by a 1 μm-thick layer of aluminum (applied by PVD). The deposit is then cured in air at 982°C to form an "alumino-borosilicate-like" glass. The multi-layer sol-gel coating is about 5 μm thick and consists of a 0.5 μm thick refractory base-coat plus a two-layer-glass oxygen protection layer. A 4 μm thick glass "A" serves as an oxygen diffusion barrier and a 0.5 μm thick glass "B", designed to be molten at the exposure temperature, functions to seal cracks and other defects in the diffusion barrier layer. For the conditions investigated here, all the coatings investigated provided some oxidation protection to the substrate. The multi-layer sol-gel coating provides the best oxidation protection for Ti-14Al-21Nb alloy and the Al-SiB_x and Sermalloy "J" coatings provide about equal protection.



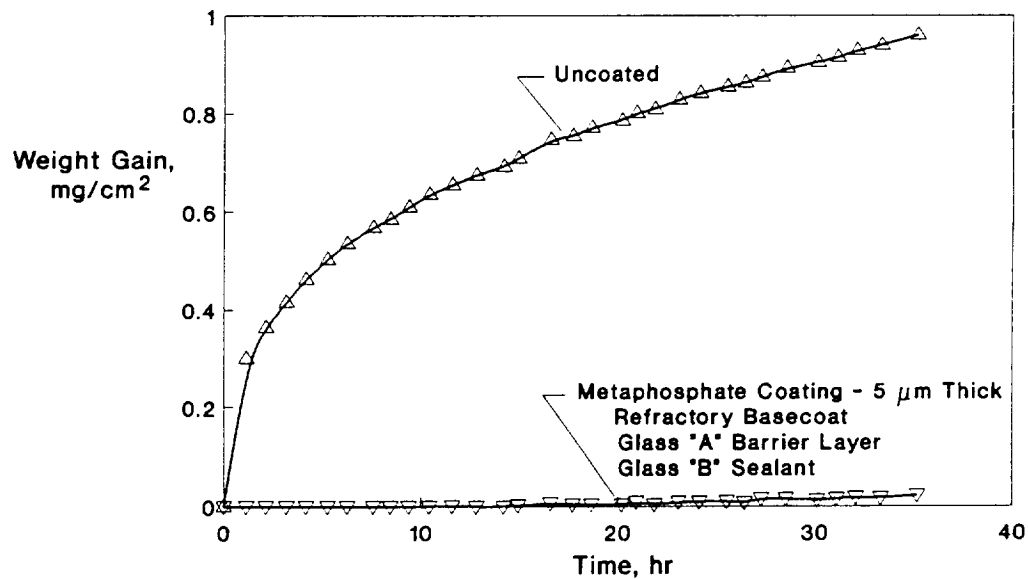
Al-SiB_x COATING: STATIC VERSUS CYCLIC OXIDATION
Ti-14Al-21Nb IN AIR AT 982°C

The Al-SiB_x coating has been evaluated under cyclic oxidation conditions. The figure shows cyclic oxidation weight gain data for 100 cycles with 15 min at temperature for each cycle. The total weight gain after 100 cycles was about 15% greater than for static oxidation exposure for the same cumulative times.



CYCLIC OXIDATION: SOL-GEL COATING ON BETA-21S ALLOY
Each Cycle Nominal 1 hr at 800°C

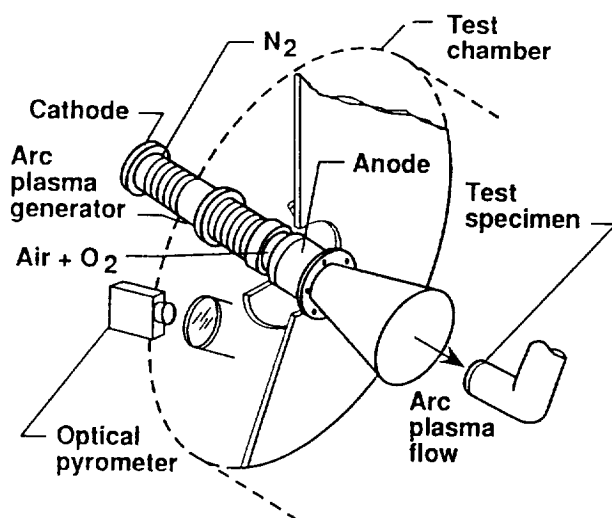
The figure shows weight gain data for 800°C cyclic oxidation tests of uncoated Beta-21S titanium alloy and the alloy with a 5 μm thick multi-layer metaphosphate coating. The data show that oxidation weight gain for the alloy with the coating was about 2% of the level for the uncoated alloy.



SCHEMATIC DIAGRAM OF LaRC HYMETS FACILITY

Thermal control/environmental coatings were tested under simulated hypersonic flight conditions at surface temperatures from 800 to 982°C in the NASA Langley Research Center Hypersonic Materials Environmental Test System (HYMETS). The HYMETS is a 100-kW constrictor-arc heated wind tunnel (Ref. 1). The test facility consists of the arc heater, a test chamber with three model insertion stings, and continuous-duty air pumps. Test samples, measuring 2.5 cm in diameter, were mounted on stagnation model adapters attached to the insertion stings. Another sting contained a water-cooled heating rate and pressure probe that measured the cold-wall heating rate and the surface pressure. The test gas was a mixture of air plus nitrogen and oxygen in ratios equivalent to air. High purity nitrogen was introduced at the cathode, and air and high purity oxygen were introduced in the plenum upstream of the supersonic nozzle. The temperature of the samples during exposure was monitored by a thermocouple attached to the back surface of the sample. The power input to the arc heater and the gas flow rate in the facility were controlled to maintain the desired sample temperature during tests.

The range of HYMETS test conditions that can be achieved are shown in the figure. These test conditions do not provide for full simulation of hypersonic flight conditions; however, the heating rate (the most critical test parameter) is representative of the levels encountered by a significant portion of a vehicle in hypersonic flight. Chemical equilibrium calculations for the operating conditions used in the present study indicate that oxygen in the test stream was almost fully dissociated (> 95%) and the nitrogen was only slightly dissociated (< 5%) (Ref. 2).

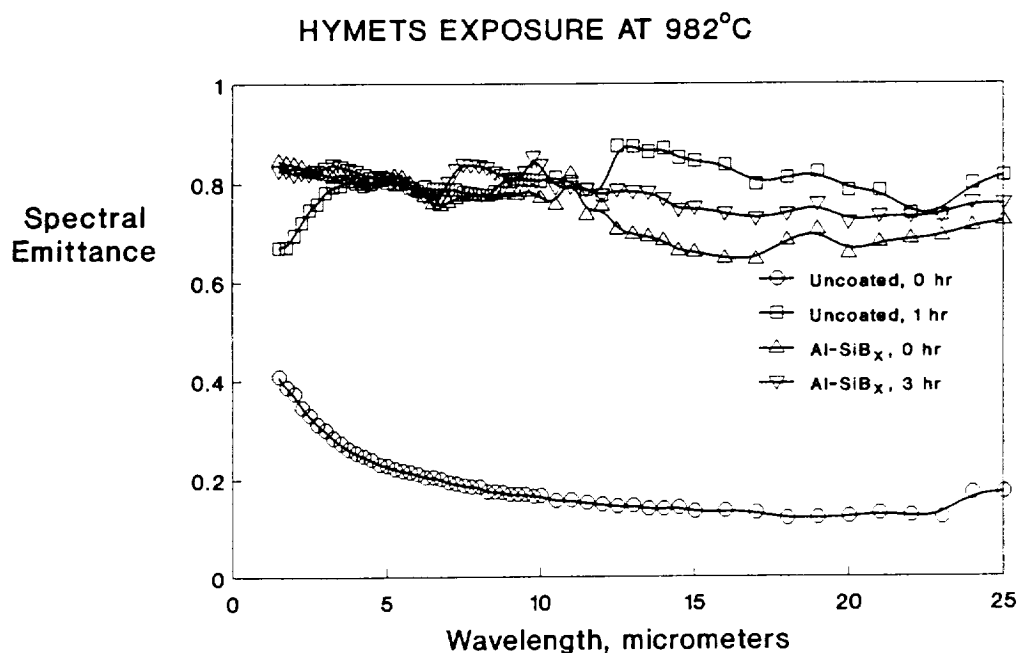


Range of test conditions

Specimen	2.5 cm diam., stagnation
Spec. Temp, °C	800 - 1500
Surface pressure, Pa	400 - 800
Free stream enthalpy, MJ/kg	4 - 11
Free stream Mach no.	3.5 - 4.0
Cold wall heating rate, kW/m ²	80 - 450

SPECTRAL EMITTANCE OF Ti₃Al-Nb ALLOY

Room-temperature spectral near-normal reflectance measurements were made over the wavelength range of 1.5 to 25 μm using a Gier Dunkle Model HCDR 3 heated-cavity reflectometer (Ref. 3). Emittance was calculated from reflectance using the following relationships: absorptance equals unity minus reflectance, and (from Kirchhoff's law) emittance equals absorptance. The figure shows spectral emittance data for coated and uncoated Ti-14Al-21Nb alloy before and after testing in the HYMETS facility. The coating is a proprietary Al-SiB_x coating developed by Lockheed Missiles and Space Company. The emittance of the coated sample was uniformly high before and after testing. The low emittance of the uncoated sample before testing is typical of the emittance of bare metal surfaces. After testing, the uncoated sample exhibited a substantially higher emittance compared to the data before testing. The increase in emittance with testing of the uncoated sample was due to the heavy oxide layer that formed at the surface during testing.



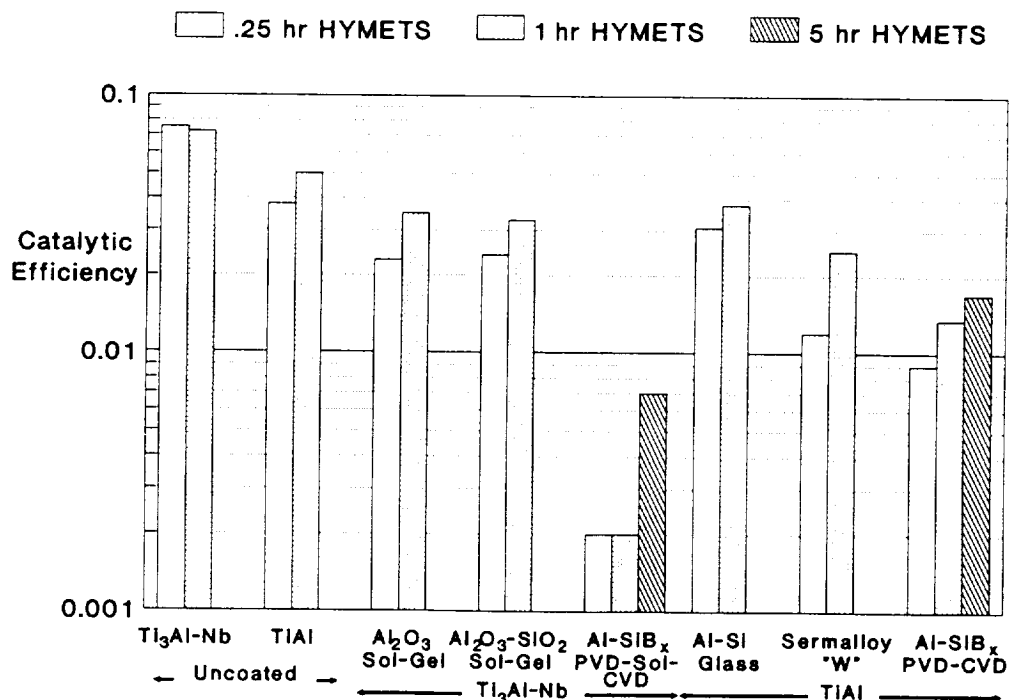
TITANIUM-ALUMINIDE ALLOYS AND COATINGS **TOTAL EMITTANCE AT 982°C**

The total normal emittance at 982°C was obtained by numerically integrating the spectral emittance data over wavelength, where the spectral data was weighted by the Planck

TITANIUM-ALUMINIDE ALLOYS AND COATINGS **CATALYTIC EFFICIENCY AT 982°C**

The catalytic efficiencies of samples under steady-state heating conditions were determined using the aerothermal heating to the sample (calculated as the sum of energy radiated from the sample and energy conducted to the sample holder) with the HYMETs operating conditions and Goulard's solution to the stagnation laminar flow heating equation (Ref. 7 and 8). For hot structures at hypersonic flight conditions, these data are more important than the room temperature catalytic efficiencies shown in the previous figure.

This figure shows a comparison of catalytic efficiencies at 982°C for uncoated Ti_3Al-Nb and $TiAl$ alloys and for six coatings. The catalytic efficiency of each of the coatings was somewhat lower than the catalytic efficiencies of the uncoated alloy samples. The lowest catalytic efficiencies were exhibited by the $Al-SiB_x$ coatings: one formed by a three step PVD-sol-CVD process and one formed by a two step PVD-CVD process. Catalytic efficiency data for other coatings were reported in Ref. 5.



CONCLUDING REMARKS

NASA Langley Research Center has devoted extensive research and development effort to thermal control and oxidation-resistant coatings for titanium-based alloys. More than 40 different alloy-coatings systems have been evaluated for potential use as environmental/thermal control applications on hypersonic vehicles. A number of the coatings evaluated have a high emittance and a low catalytic efficiency compared to the uncoated alloys. The most attractive thermal control coating examined to date is an Al-SiB_x coating: its emittance is high and its catalytic efficiency is the lowest measured to date; with a thickness of $5\text{ }\mu\text{m}$, it compares favorably with commercially available oxidation protection coatings that are up to $50\text{ }\mu\text{m}$ in thickness; and it is applied by processes that can be scaled-up to coat large structures.

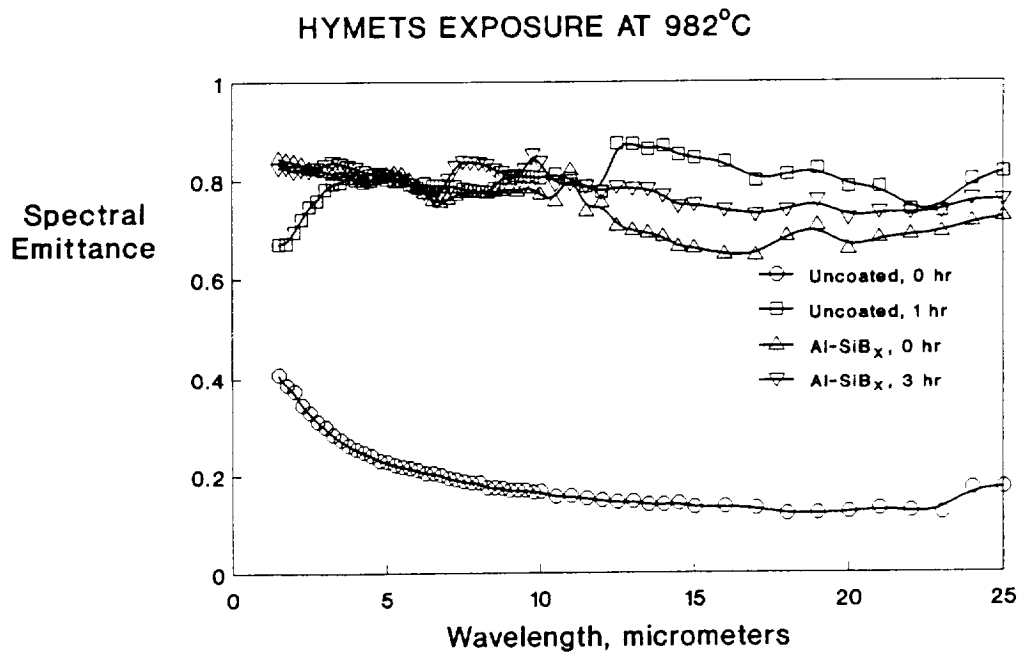
NASA LaRC has made significant progress in development of oxidation protection coatings based on sol-gel processes. The oxidation protection by a $5\text{ }\mu\text{m}$ -thick multi-layer coating prepared by sol-gel processes is superior to any other coating evaluated to date.

REFERENCES

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8. Wiedemann, K. E.; Clark, R. K.; and Sankaran, S. N.: Emittance, Catalysis, and Dynamic Oxidation of Ti-14Al-21Nb. NASA TP-2955, 1989.

SPECTRAL EMITTANCE OF Ti₃Al-Nb ALLOY

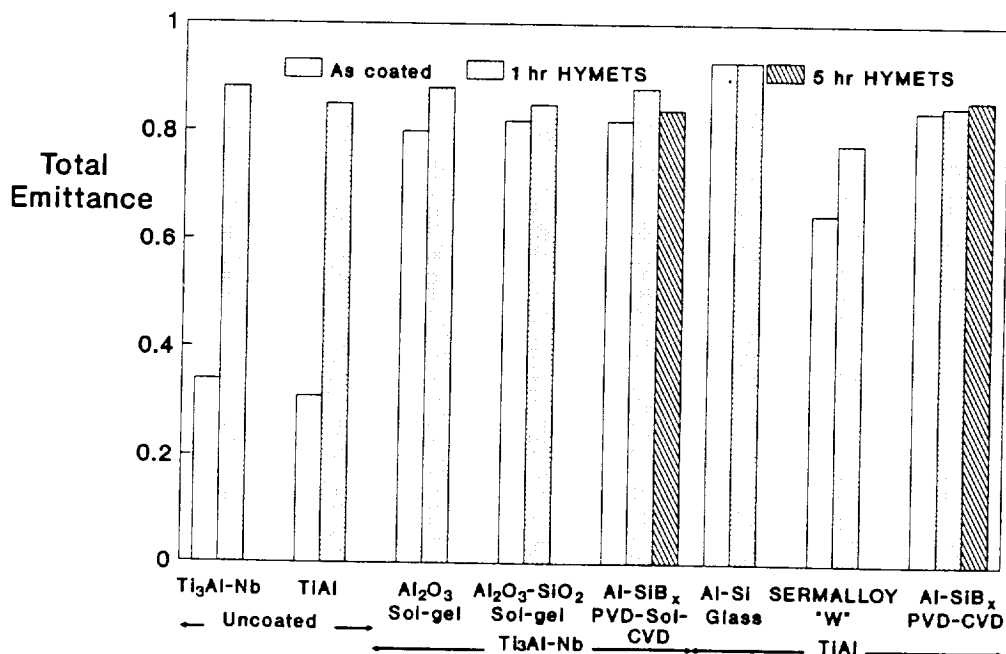
Room-temperature spectral near-normal reflectance measurements were made over the wavelength range of 1.5 to 25 μm using a Gier Dunkle Model HCDR 3 heated-cavity reflectometer (Ref. 3). Emittance was calculated from reflectance using the following relationships: absorptance equals unity minus reflectance, and (from Kirchhoff's law) emittance equals absorptance. The figure shows spectral emittance data for coated and uncoated Ti-14Al-21Nb alloy before and after testing in the HYMETS facility. The coating is a proprietary Al-SiB_x coating developed by Lockheed Missiles and Space Company. The emittance of the coated sample was uniformly high before and after testing. The low emittance of the uncoated sample before testing is typical of the emittance of bare metal surfaces. After testing, the uncoated sample exhibited a substantially higher emittance compared to the data before testing. The increase in emittance with testing of the uncoated sample was due to the heavy oxide layer that formed at the surface during testing.



TITANIUM-ALUMINIDE ALLOYS AND COATINGS **TOTAL EMITTANCE AT 982°C**

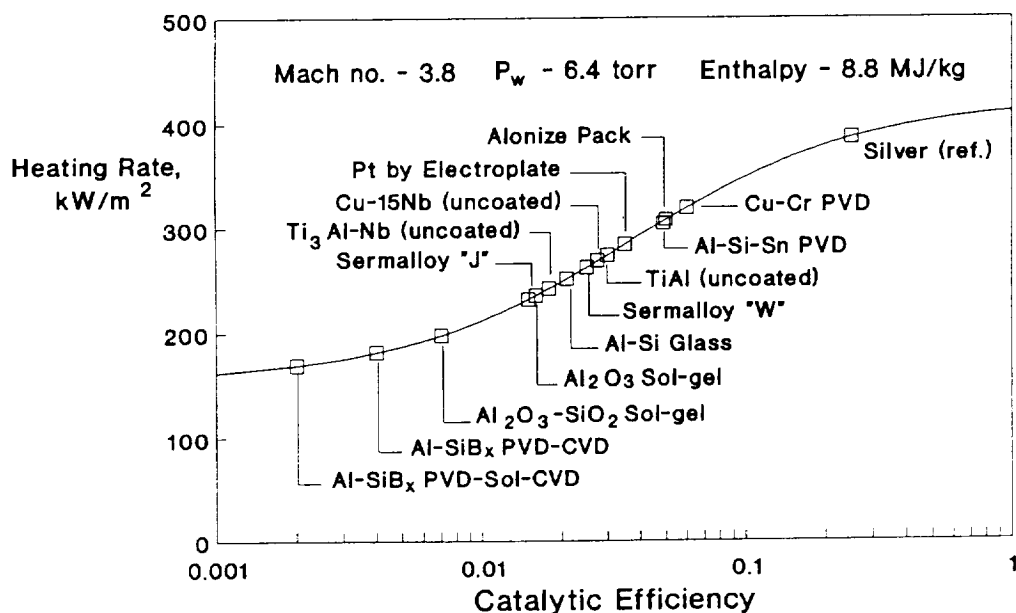
The total normal emittance at 982°C was obtained by numerically integrating the spectral emittance data over wavelength, where the spectral data was weighted by the Planck blackbody energy distribution function at 982°C (Ref. 4). The figure shows a comparison of total normal emittance of uncoated Ti₃Al-Nb and TiAl alloys and six coatings of current interest. Data are shown for two sol-gel coatings (Al₂O₃ and Al₂O₃-SiO₂), a glass slurry coating (Al-Si), a commercially available reactive coating (Sermalloy "W"), and two Al-SiB_x coatings (one produced by a three-step PVD-Sol-CVD process and one by a two-step PVD-CVD process).

The total emittance of the uncoated alloys after HYMETS testing was significantly higher than before testing. The Sermalloy "W" coated TiAl sample had a lower emittance than did the remaining coatings: in fact the Sermalloy "W" coating did not meet the performance requirement goal of 0.8 for emittance of thermal control coatings for hypersonic applications. Emittance data for other candidate thermal control coatings were reported in Ref. 5.



VARIATION IN COLD-WALL HEATING WITH CATALYTIC EFFICIENCY

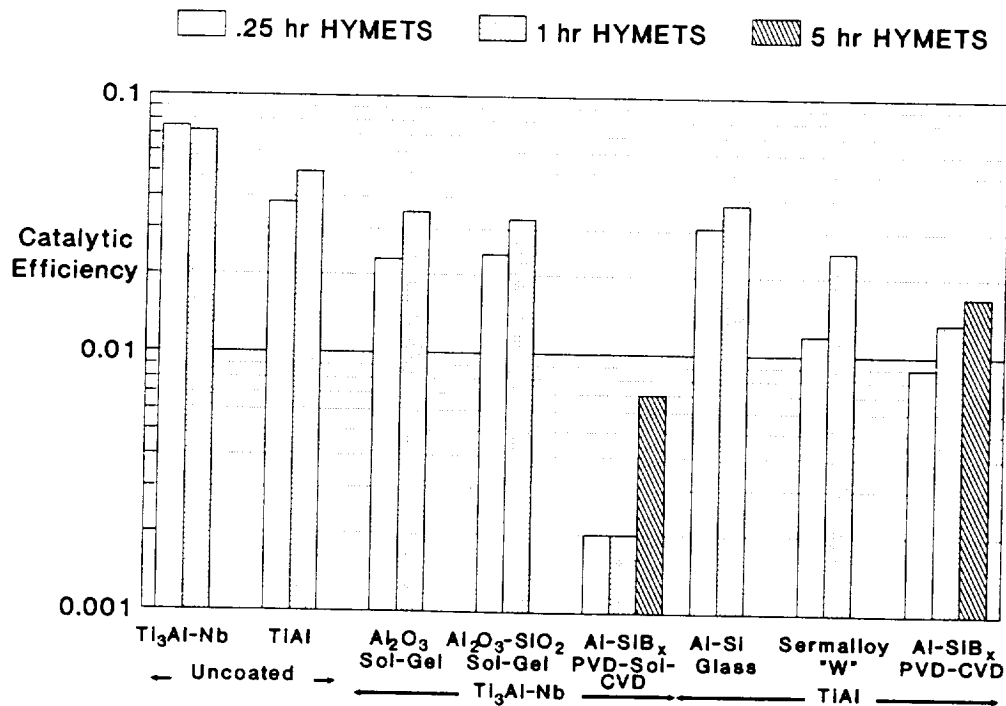
The figure shows the variation in cold-wall heat transfer with catalytic efficiency for a sample exposed to a Mach 3.8 flow with a total enthalpy of 8.8 MJ/kg and a wall pressure of 6.4 torr. For non-catalytic surfaces (very low catalytic efficiencies) the heating is almost totally due to convective heat transfer. The data show that, for this test condition, the catalytic heating (heating due to recombination of dissociated oxygen and nitrogen at the surface) to a fully catalytic surface is greater than the convective heating. As a point of reference, for a hypersonic vehicle operating at a surface temperature of 1175°C, a change in the heating rate from 200 kW/m² to 300 kW/m² could increase the equilibrium surface temperature by about 165°C. This figure also presents representative data for a number of coatings and for uncoated Ti₃Al-Nb, TiAl, and Cu-Nb alloys. Silver, which has a catalytic efficiency of 0.25 (Ref. 6), was used as a reference. The catalytic efficiencies of uncoated samples and some metallic coatings were somewhat lower than the catalytic efficiency of silver. The Al-SiB_x coatings and the Al₂O₃-SiO₂ coating had much lower catalytic efficiencies than the remaining coatings and the uncoated samples. Catalytic efficiency data for other coatings were reported in Ref. 5.



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